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Im Auftrag

For the President of the European Patent Office  
Le Président de l'Office européen des brevets  
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Polyamine derivatives, a process to make them, and their use

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## POLYAMINE DERIVATIVES, A PROCESS TO MAKE THEM, AND THEIR USE

The invention relates to a process to make polyamine derivatives with at least one anchoring group and at least one matrix-compatible moiety, the polyamine derivatives so obtainable, and the use of such polyamine derivatives in conventional applications, such as pigment dispersants in inks, wetting agents in coating compositions, and transfer resins in pigment processing operations.

EP-A-0 358 358 disclosed the use of specific amine derivatives as pigment dispersing agents. However, such conventional polyester based pigment dispersants (see also WO 98/19784) often lead to crystallization problems, particularly when used in acryloyl functional acrylic resin-based compositions. Acryloyl functional resin-based coating compositions become more popular since they are very useful in coating compositions which are cured by at least a UV-curing step. Furthermore, there is a need for alternative agents for non-UV cure applications, including printing inks, varnishes, and non-UV cure coating compositions. The alternative agents preferably have better dispersing properties than the conventional pigment dispersants and preferably are suitable in virtually all conventional applications, without that any further processing steps, such as the re-heating of the formulation to (re)dissolve any precipitate, are required.

The present invention relates to such alternative agents. In a preferred embodiment it relates to specific polyamine derivatives containing two or more matrix-compatible moieties.

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Surprisingly, we found a process to make polyamine derivatives which are very versatile and typically will outperform the products of EP-A-0 358 358 (WO 98/19784) in pigment dispersing operations, particularly since the anchoring

moiety of the polyamine derivatives has improved interaction with pigments, while the matrix-compatible moiety can be tailored to ensure full compatibility of the derivatives and the matrix in which they are used, particularly in combination with acryloyl functional resins. Accordingly, we claim the process to make these 5 polyamine derivatives, intermediates of the process, the products obtainable, and their use.

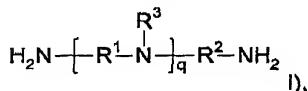
The process according to the Invention is characterised in that one or more polyamines, each with one or more  $-\text{NH}_2$  functions and one or more second amine 10 functions, said second amine functions having a lower lactone reactivity than said  $-\text{NH}_2$  functions and whereby each second amine function, independently, can be primary or secondary, is reacted in a first step with one or more lactones, cyclic carbonates, hydroxyacids, or mixtures thereof, to form a polyamine-derived compound comprising unreacted second amine function; amide and/or urethane 15 groups; optionally ester and/or carbonate groups; and one or more  $-\text{OH}$  groups, which compound is reacted in a second step with an at least bifunctional amine-specific reagent to form an intermediate comprising at least two polyamine residues with amide and/or urethane groups and optional ester and/or carbonate groups that are linked by said amine-specific reagent residue. If so desired, any 20 conventional catalyst can be used in either the first or second step. The molecular ratio in which the polyamines are reacted with said lactones, cyclic carbonates, hydroxyacids, or mixtures thereof, can vary over a wide range. Preferably on average at least 0.1, more preferably at least 0.5, most preferably at least 1 lactone, cyclic carbonate, and/or hydroxyacid molecule is reacted per  $-\text{NH}_2$  25 function in the reaction mixture. Similarly, preferably at most 10, more preferably at most 5, and most preferably at most 3 cyclic carbonate, and/or hydroxyacid molecule reacts on average per  $-\text{NH}_2$  function.

The at least two linked polyamine residues with amide and/or urethane groups and optional ester and/or carbonate groups were found to form an anchoring group with very good interaction with pigments and plastic surfaces. Depending on the molar ratio of reactants in the first step and the nature of the at least bifunctional 5 amine-specific reagent it is possible to tailor the anchoring group to optimize this interaction. Optionally, but less preferred, the process comprises an intermediate step wherein the -OH groups, or parts of the unreacted amine groups, are reacted before the step wherein the polyamine residues are linked with the at least bifunctional amine-specific reagent. If this intermediate step is performed, then 10 care is to be taken that the second amine functions survive the reaction step, in order to allow the linking to occur.

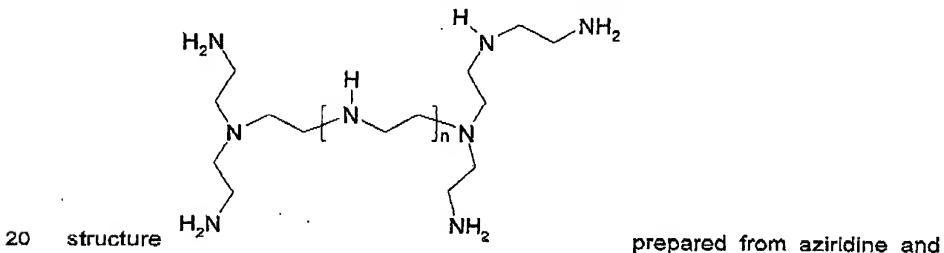
The polyamines that are used in accordance with the invention are substituted or unsubstituted, linear or branched, hydrocarbons containing one or more -NH<sub>2</sub> 15 groups and one or more second amine functions with a lower lactone reactivity than said -NH<sub>2</sub> functions. Although less preferred, these polyamines may also possess other less reactive groups, e.g. hydroxyl groups. To establish whether or not the amine groups of the polyamine have the necessary difference in reactivity towards lactones and/or cyclic carbonates, one can start from common general 20 knowledge with respect to the reactivity of amines, including the knowledge that, In such reactions, primary amines are more reactive than secondary amines, and primary amines on secondary carbon atoms are more reactive than primary amine groups on tertiary carbon atoms. Helpful is the simple test wherein 1 mole of - caprolactone is reacted with a mixture comprising 1 mole of a low molecular weight mono- 25 amine analogue with said -NH<sub>2</sub> group and 1 mole of a low molecular weight mono- amine analogue with said second amine function. The reaction can be performed with or without solvents at a temperature where reactivity is observed which can be monitored with e.g. a titration on amines. Suitably the reaction is conducted at

100°C for a period of 2 hours. Low molecular weight compounds are suitably compounds with a molecular weight of from 50 to 150 Dalton. In the test but, if this can be analysed, preferably in the reaction of the polyamine, at least 60%, on a molar basis, of the lactone, hydroxy acid, and/or cyclic carbonate reacts with the –NH<sub>2</sub> function of the polyamine. More preferably at least 80%, on a molar basis, of the lactone, hydroxyacid and/or cyclic carbonate reacts with the –NH<sub>2</sub> function and most preferably at least 90%, on a molar basis, of the lactone, hydroxyacid, and/or cyclic carbonate reacts with the –NH<sub>2</sub> function of the polyamine.

10 Preferred polyamines are compounds of the formula I),



wherein q is an integer from 1 to 10, preferably 1 to 5, more preferably 1 or 2, R<sup>1</sup> and R<sup>2</sup>, independently, represent alkylene groups with from 1 to 10 carbon atoms, preferably from 1 to 6 carbon atoms, and each of R<sup>3</sup> being independently selected from hydrogen, alkyl, alkylamine, polyalkylamine, and polyalkylpolyamine. Preferably R<sup>1</sup> and R<sup>2</sup> are the same. Examples of products according to formula II) include: diethylene triamine (q=1), triethylene tetramine (q=2), tetraethylene pentamine (q=3), triethylene tetramine (q=1, R<sup>3</sup> is ethylamine), dipropylene triamine (q=1), dihexylene triamine (q=1), and compounds of the general branched



known as polyethylenimine and commercially available under the tradename Epomin® SP with Mw's up to 10,000 or similar, e.g. compounds having propylene rather than ethylene moieties, or compounds with more or less primary, secondary, or tertiary amine groups. The compounds can also comprise a combination of 5 ethylene, propylene and/or hexylene units.

The lactone, hydroxyacid, and/or cyclic carbonate can be selected from any conventional lactone, hydroxyacid, and/or cyclic carbonate, including, but not limited to, butyrolactones, valerolactones, caprolactones, ethylene carbonate, 10 propylene carbonate, glycerol carbonate, hydroxy stearic acids, hydroxybutyric acids, and hydroxycaprylic acid. Preferably a conventional lactone is selected. The lactones can be of the beta-, gamma-, delta-, and/or epsilon-type. In view of their stability and availability, gamma-, delta- and epsilon-lactones are most preferred.

15 The product of the first step is reacted with an at least bifunctional amine-specific reagent. With amine-specific is meant, that if the polyamine derived compound comprises both the second amine function and -OH groups, then at least 60%, on a molar basis, of the amine-specific reagent groups react with the second amine function. Preferably, at least 80%, most preferably at least 90%, both on a molar 20 basis, of the amine-specific reagent groups react with the second amine function of the polyamine derived compound. Preferably, the specificity of the reaction is measured by analysing the reaction product of the process according to the invention. However, if this is not feasible, then a simple test method can be used wherein a mixture comprising an equal molar amount of a low molecular weight 25 mono-OH-functional analogue and a low molecular weight analogue compound with just one second amine function is reacted in a suitable solvent at a temperature of 80°C, optionally in the presence of a solvent, until at least 80%, preferably at least 90% of the amine has reacted with the amine-specific reagent,

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after which the specificity of the reaction can be determined using conventional techniques.

The amine-specific reagent is typically used in an amount wherein the amount of amine-reactive groups corresponds from 0.1 to 10 times the amount of second 5 amine function groups of the polyamine-derived compound. In a preferred embodiment, the amine-specific reagent is used in an amount wherein the amount (in equivalents) of amine-reactive groups corresponds from 1 to 10 times the amount (in equivalents) of second amine function groups of the polyamine-derived compound, particularly if the amine-specific reagent comprises three or more 10 reactive groups. Preferred amine-specific reagents include isocyanates, anhydrides, acid chlorides, epoxy compounds, maleates, fumarates, citraconic esters, itaconic esters, and (meth)acrylates. More preferably the amine-specific reagent is a conventional di-, or poly-isocyanate, including compounds like 15 hexylene-1,6-diisocyanate, isophorone diisocyanate, toluene diisocyanate, and oligomeric or polymeric diphenyl methane diisocyanate (MDI). It is to be understood that if the amine-specific reagent comprises more than two amine-reactive groups, then these additional groups of R<sup>3</sup> may or may not have reacted with amine functions of the same or other polyamine-derived compound molecules.

20

The intermediate from the second reaction step comprises -OH groups, which are reacted in a third step of a preferred process with compounds that introduce a matrix-compatible moiety. With matrix-compatible is meant that said moiety has affinity for the continuous medium in which the final polyamine derivative is to be 25 used as dispersant, wetting agent, or the like. Preferably, the polyamine derivative are used in coating compositions and printing inks, most preferably in acryloyl-functional resin-containing compositions. Non-aqueous coating compositions and printing inks are preferred. The matrix-compatible moiety has a molecular weight of

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more than 250, preferably more than 500, more preferably more than 800, even more preferably more than 1200 and, most preferably, more than 2000 Dalton, whereby the molecular weight preferably is below 20.000, more preferably below 10.000 Dalton. If the polyamine derivative is to be used in aqueous compositions,

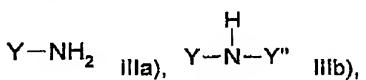
5 e.g. as a paint dispersant in water-borne coating compositions, then the third step preferably comprises an alkoxylation process. Said alkoxylation process can comprise one or more ethoxylation and/or propoxylation steps, in any order or sequence.

appropriate

10 If the polyamine derivative is to be used in non-aqueous compositions, then at least part of the OH groups of the intermediate is preferably reacted with an epoxide; lactone; cyclic carbonate; (hydroxy)acid, ester, anhydride, etc., to substitute a linear or branched, substituted or unsubstituted, preferably unsubstituted,  $C_4$ - $C_{30}$  alkyl, polyester, polyether, polyetherester and/or 15 polyesterether group for the proton.

It should be noted that the anchoring moiety can be modified during any time of the process, provided that the final product resulting from the process bears at least one essential anchoring group and at least one of said matrix-compatible 20 moieties. Two examples of preferred modifications are the following:

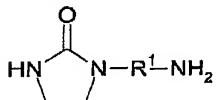
1.) in the second step of the process, at least one additional amine is introduced of the formulae IIIa) and/or IIIb),



wherein Y represents a moiety with affinity for a pigment surface or plastic article 25 and Y'' is selected from the group consisting of groups Y, linear or branched, substituted or unsubstituted, preferably unsubstituted,  $C_4$ - $C_{30}$  alkyl, polyester, polyether, polyetherester and polyesterether. More preferably, group Y is an

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aliphatic group with from 2 to 10 carbon atoms containing at least one tertiary amino group or a heterocyclic group containing at least one basic ring nitrogen atom and where the heterocyclic group may be attached to the NH group (short for the -NH<sub>2</sub> group of formula IIIa or the -NH- group of formula IIIb) by an alkylene group containing up to 10 carbon atoms. Preferably, Y is a mononuclear or dinuclear heterocyclic group having a ring nitrogen atom which is attached to the NH group, preferably by a C<sub>2-5</sub>-alkylene. Preferred heterocyclic groups are optionally substituted triazole, pyrimidine, imidazole, pyridine, morpholine, pyrrolidine, piperazine, benzimidazole, benzthiazole and/or triazine. Substituents 5 may be C<sub>1-6</sub> and especially C<sub>1-4</sub>-alkyl or alkoxy or amino. As noted hereinbefore Y may be attached to the NH group via an alkylene group containing up to 10 carbon atoms, preferably it is a C<sub>2-5</sub>-alkylene and more preferably a C<sub>2-4</sub>-alkylene group. Y may also be attached to the NH group via a polyether group containing the same 10 number of carbon atoms as the alkylene group. Examples of preferred products of formulae IIIa) and IIIb) are: nitrogen-substituted alkanediamines with an NH<sub>2</sub> group and a secondary or tertiary amine function, 1-(2-aminoethyl)- piperazine, 2-(1-pyrrolidyl)-ethylamine, 4-amino-2-methoxypyrimidine, products of formula IV), 15

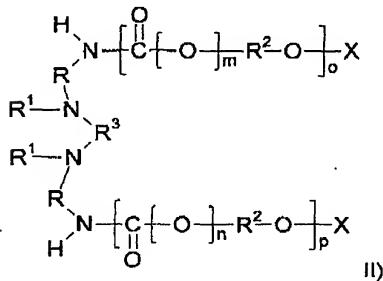


IV), and Jeffamines®. More preferably, the 20 compounds are selected from the group consisting of N, N-dimethyl-1,3-propanediamine, 4-(2-aminoethyl)-pyridine, 2-amino-6-methoxybenzothiazole, 4-(aminomethyl)-pyridine, N,N-diallylmelamine, 3-amino-1,2,4-triazole, 1-(3-aminopropyl)-imidazole, 3-mercaptop-1,2,4-triazole, products of formula III), and products of formula IV). The additional amine was found to be incorporated into the anchoring group, allowing further tailoring. The amount of at least bifunctional

amine-specific reagent in step 2 is to be adjusted such that sufficient bifunctional amine-specific reagent is present to also react with the additional amine.

2.) similarly to modification 1, at least one monofunctional amine-specific reagent of formula Z-A is present in step 2 of the process, wherein Z is selected from the 5 group consisting of linear or branched, substituted or unsubstituted, preferably unsubstituted, C<sub>4</sub>-C<sub>30</sub> alkyl, polyester, polyether, polyetherester and polyesterether, and A is an amine-specific reactive group as defined for step 2 of the process. The additional monofunctional amine-specific reagent was found to be attached to the anchoring group, allowing further tailoring of the final polyamine derivative, to 10 optimize its for the use in various media, using varying pigments and/or plastic substrates. The amount of at least bifunctional amine-specific reagent in step 2 may be adjusted such that the total amount of reactive groups of the at least 15 bifunctional amine-specific reagent and the monofunctional amine-specific reagent is within the range of 1 to 10 times the amount of second amine function groups of the reaction mixture.

In another preferred embodiment, the invention also relates to compounds obtainable by the above-identified process which comprise said anchoring and matrix-compatible moieties. Such compounds can be represented by the formula 20 II):



wherein each  $R^1NRNH$  moiety is a residue of the polyamine that was used, each  $C(O)[O]R^2O$  moiety is a residue from the lactone, hydroxyacid, and/or cyclic carbonate that was used, and  $R^3$  is the residue of the at least bifunctional amine-specific reagent. More particularly, each  $R$  and/or  $R^1$  may, independently, be  
5 substituted with further primary or secondary amine groups, each of which may or may not have reacted with further lactone, hydroxyacid, and/or cyclic carbonate.  $R^2$  represents a linear or branched, substituted or unsubstituted, hydrocarbon. Depending whether or not a lactone, hydroxyacid or a cyclic carbonate is used,  $n$  and  $m$  will be 0, 0, or 1, respectively. The indices  $\sigma$  and  $\rho$  present the average  
10 number of moiety  $C(O)[O]R^2O$  per  $R^1NRNH$  moiety and have a value ranging from 0.1 to 10, preferably 0.5 to 5, most preferably 1 to 3. For the molecules as derived in the above-mentioned process, each  $X$  is preferably hydrogen. However, if the optional intermediate step as presented above is taken or if the  $-OH$  group is reacted in later steps, then  $X$  is, wholly or partly, a substituted or unsubstituted,  
15 linear or branched, hydrocarbon group.

The moiety of formula II), minus groups  $X$ , was found to be a very suitable anchoring moiety for interaction with a variety of pigments and substrates. However, in order to make a good dispersant, the moiety is to be combined with at  
20 least one matrix-compatible moiety to form suitable dispersants, wetting agents, compatibilisers, and the like. Accordingly, each group  $X$  is, independently, preferably such a matrix-compatible moiety. Typically, this is achieved by reacting the product of formula II) wherein  $X$  is hydrogen with a suitable compound. Preferably, each of groups  $X$ , independently, is a compound with a molecular  
25 weight at more than 250, preferably more than 500, more preferably more than 800, even more preferably more than 1200 and, most preferably, more than 2000 Dalton, whereby the molecular weight of group  $X$  preferably is below 20.000, more preferably below 10.000 Dalton. Most preferably the groups  $X$  are fully compatible

with binder resins of coating composition in which the products according to the invention are used.

In a preferred embodiment the groups X are selected such that the final compound  
5 is fully compatible with acryloyl functional resins. Preferably groups X are, independently, selected from linear or branched, substituted or unsubstituted, preferably unsubstituted, C<sub>4</sub>-C<sub>30</sub> alkyl, polyester, polyether, polyetherester or polyesterether groups. Preferably groups X are formed by reacting a compound of formula II) wherein X is hydrogen with appropriate epoxides; lactones; cyclic  
10 carbonates; hydroxyacids, and other suitable conventional reactants to form polyesters, e.g. via an alternating co-polymerisation; and polyethers. Alternatively, a linear or branched, substituted or unsubstituted, preferably unsubstituted, C<sub>4</sub>-C<sub>30</sub> alkyl, polyester, polyether, polyetherester or polyesterether with OH reactive groups is reacted with compound II) with X is hydrogen. If so desired, conventional  
15 catalysts can be used in this step. Especially good pigment dispersants were obtained when the -OH function of the polar moiety is reacted in an alternating co-polymerization with hexahydrophthalic anhydride and Cardura® E10, to form a polyester function.

20 The invention is elucidated by the following examples.

Example 1

**a) Preparation of the amide-amine:**

In a three-necked roundbottomed flask equipped with a mechanical stirrer and a reflux condenser, 61.85 grams (0.60 mole) of diethylenetriamine (ex. Aldrich) and 25 136.85 grams (1.12 mole) of -caprolactone (ex. Acros) were weighed. The reaction mixture was heated on an oil bath, at 140°C, for 10h to complete the reaction. A polyamine-derived compound was obtained.

**b) Preparation of the tetrahydroxy-tetraamide-diurea**

In a 250ml three-necked round-bottomed flask equipped with a mechanical stirrer, and a reflux condenser, 96.90 grams (0.292 mole) of the polyamine-derived compound of Example 1a was weighed. Temperature was raised to 65°C and 24.50 grams (0.146) of hexamethylene diisocyanate (ex. Fluka) was added over a period of 45 minutes while keeping the reaction mixture at 65°C. Temperature was next raised to 100°C in order to complete the reaction of forming a tetrahydroxy-tetraamide-diurea.

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**c) Preparation of a pigment dispersant with a polyester tail**

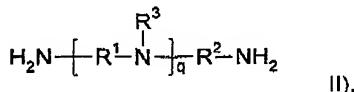
In a 500ml three necked round-bottomed flask equipped with a reflux condenser, mechanical stirrer and a thermometer 40.21 grams (48.4 mmole) of the tetrahydroxy-tetraamide-diurea of Example 1b was weighed together with 132.75 grams (0.861 mole) of hexahydrophthalic anhydride (ex. Lonza) and 0.39 grams of a chromium based catalyst (Accelerator AMC-2, ex. Flevo Chemie). The reaction mixture was heated to 155°C. Next 215.26 grams (0.878 mole) of Cardura® E10 (ex. Resolution) was added over a period of 2.5h. An additional hour completed the reaction.

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The product of Example 1c showed very good pigment dispersant properties, had a desirable low viscosity, higher gloss, and was fully compatible with an acrylated resin, therefore no precipitate (crystals) were formed, allowing the use of the formulation "as is", meaning that it does not require re-heating before use. It was found to be an improvement over conventional products, also because the polyamine derivative was easily dissolved in the resin while it allowed for a much easier dispersion of the pigment into the formulation.

CLAIMS

1. A process wherein one or more polyamines, each with one or more -NH<sub>2</sub> functions and one or more second amine functions, said second amine functions having a lower lactone reactivity than said -NH<sub>2</sub> functions, is reacted in a first step with one or more lactones, hydroxyacids, cyclic carbonates, or mixtures thereof, to form a polyamine-derived compound, which compound is reacted in a second step with one or more at least bifunctional amine-specific reagents to form an intermediate comprising at least two polyamine residues with amide and/or urethane groups and optional ester and/or carbonate groups that are linked by said amine-specific reagent residue.  
5
2. Process according to claim 1 wherein the amount of lactone, hydroxyacid and/or cyclic carbonate molecules is from 0.1 to 10 times the amount of -NH<sub>2</sub> groups of the polyamine.  
15
3. A process according to claim 1 or 2 wherein the amine-specific reagent is used in an amount such that the amount of amine-specific reagent groups corresponds to from 0.1 to 10 times the amount of second amine function groups of the polyamine-derived compound.  
20
4. A process according to any one of the preceding claims wherein a polyamine is used of the formulae II)



25 wherein q is an integer from 1 to 10, wherein R<sup>1</sup> and R<sup>2</sup>, independently, are selected from alkylene groups with from 1 to 10 carbon atoms, and wherein

each of  $R^3$ , independently, is selected from the group consisting of hydrogen, alkyls, alkylamines, polyalkylamines, and polyalkylpolyamines.

5. A process according to any one of the preceding claims which comprises a further step wherein one or more of the  $-OH$  groups which are formed in step 1 are reacted as to attach a matrix-compatible moiety with a molecular weight at more than 250 to said intermediate, with said further step being conducted either between steps 1 and 2 or, preferably, after step 2.
- 10 6. A process according to claim 5 wherein the  $-OH$  groups are reacted with one or more compounds selected from the group consisting of epoxides, lactones, cyclic carbonates, hydroxyacids, and other suitable conventional reactants to form polyesters, to form matrix-compatible linear or branched, substituted or unsubstituted, preferably unsubstituted,  $C_4-C_{30}$  alkyl, polyester, polyether, 15 polyetherester or polyesterether groups.
7. A process according to any one of the preceding claims a modifying agent is present in step 2, which modifying agent is selected from monofunctional amine-specific reagents and amines of the formulae  $Y-NH_2$  and  $Y-N^H-Y''$ , 20 wherein  $Y$  represents a moiety with affinity for a pigment surface or plastic article and  $Y''$  is selected from the group consisting of groups  $Y$ , linear or branched, substituted or unsubstituted, preferably unsubstituted,  $C_4-C_{30}$  alkyl, polyester, polyether, polyetherester and polyesterether.
- 25 8. Products obtainable by a process according to any one of the preceding claims.

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**9. Use of the products of claim 8 in printing ink formulations.**

**10. Use of the products of claim 8 in coating compositions.**

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**Abstract**

The invention relates to processes wherein one or more polyamines, each with one or more -NH<sub>2</sub> functions and one or more second amine functions, said second 5 amine functions having a lower lactone reactivity than said -NH<sub>2</sub> functions, is reacted in a first step with one or more lactones, hydroxyacids, cyclic carbonates, or mixtures thereof, to form a polyamine-derived compound, which compound is reacted in a second step with one or more bifunctional amine-specific reagents to form an intermediate comprising at least two polyamine residues with amide and/or 10 urethane groups and optional ester and/or carbonate groups that are linked by said amine-specific reagent residue. The OH groups that are formed in the first step are reacted in a step between said steps 1 and 2 or after said step 2.